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AASERT-94 CHEMISTRY INVOLVING THE PREPARATION, ISOLATION, AND IMMOBILIZATION OF NANOCRYSTALLINE AND/OR MICROCRYSTALLINE BORON ARSENIDE, BORON PHOSPHIDE, & BORON ANTIMONIDE

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13. ABSTRACT (Maximum 200 words)

During this report period, publications 1 and 2 (see below) appeared in print (the contents of the papers were reported in two previous "Interim Technical Reports"). Michael Lube prepared the adduct $C13B.Sb(Sb(SiMe3)3)$, the third Lewis acid-base adduct of boron and antimony to be structurally characterized. This adduct along with $Br_3B.Sb(SiMe3)3$ and $I3B.Sb(SiMe3)3$ are the subject of publication 3 (see below) with the manuscript being written by Michael. Based on the various data obtained, it appeared that the black powders obtained from the thermolysis of these adducts were a mixture of nanocrystalline $Sb(hex)$ and amorphous BSb (data summarized in Michael's PhD. dissertation, 9/6/96, Duke University). Further investigations, on halo-boron-arsenic systems by Michael, with some assistance from Richard Jouet, (see publication 4 below) resulted in the isolation and structural characterization of $I3B.As(SiMe3)3$, as well as the isolation of X-ray quality crystals of the $C13B.As(SiMe3)3$ and $[I2BAs(SiMe3)2]2$ (preparations previously reported in the 1996 "Interim Technical report"). Additional studies by Richard involving $(Et_2O)_2Li(U-P(SiMe3)2)_2BH_2$ showed that (1) in a 1:1 mole ratio reaction with BC_13 , both known $[Cl_2GaP(SiMe3)2]_3$ and new $C_12Ga[U-P(SiMe3)2BH_2]$ were produced, (2) from a 1:1.25 mole ratio reaction with

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F49620-94-1-0324, Dr Well, Duke University

MeBC12, both $(Me_3Si)_2P(H)Ga[U-P(SiMe_3)_2]_2GaH_2$ and $(Me_3Si)_2P(H)Ga[U-P(SiMe_3)_2]_2B(H)Me$ could be isolated (cocrystallized in a 1:1 mole ratio), and (3) the 1:2.8 mole ratio reaction with MeBC12 afforded $(Me_3Si)_2P(Cl)Ga[U-P(SiMe_3)_2]_2B(H)Me$ as the only species to crystallize from solution. In addition, he found that the known trimer $(H_2GaP(SiMe_3)_2)_3$ was the only compound that could be isolated and characterized from the 1:1 mole ratio reactions of $(Et_2O)_2LiU-P(SiMe_3)_2]_2GaHe$ with (1) Me₂BBr and (2) H₂BC1.SMe2.

Final Technical Report

AFOSR AASERT Grant Number: F49620-94-1-0324

Research Title: *Chemistry Involving the Preparation, Isolation, and Immobilization of Nanocrystalline and/or Microcrystalline Boron Arsenide, Boron Phosphide, and Boron Antimonide*

Period Covered: 01 July 1996 - 31 Dec. 1997 (no-cost extension for the period 01 July 1997 - 31 Dec. 1997)

Principal Investigator: Professor Richard L. Wells, Department of Chemistry, Box 90346
Duke University Durham, NC 27708-0346

Graduate Students Supported: Michael S. Lube, July 1994-Sept. 1996 (completed requirements for the Ph.D. degree September 6, 1996; currently employed by IBM)

Richard J. Jouet, July 1996-Dec. 1997 (third-year graduate student; grades satisfactory; passed Ph.D. preliminary examination March 10, 1997)

Brief Narrative Report of Research Results: During this report period, publications 1 and 2 (see below) appeared in print (the contents of the papers were reported in two previous "Interim Technical Reports"). Michael Lube prepared the adduct $\text{Cl}_3\text{B}\bullet\text{Sb}(\text{SiMe}_3)_3$, the third Lewis acid-base adduct of boron and antimony to be structurally characterized. This adduct along with $\text{Br}_3\text{B}\bullet\text{Sb}(\text{SiMe}_3)_3$ and $\text{I}_3\text{B}\bullet\text{Sb}(\text{SiMe}_3)_3$ are the subject of publication 3 (see below), with the manuscript being written by Michael. Based on the various data obtained, it appeared that the black powders obtained from the thermolysis of these three adducts were a mixture of nanocrystalline $\text{Sb}(\text{hex})$ and amorphous BSb (data summarized in Michael's Ph.D. dissertation, 9/6/96, Duke University). Further investigations on halo-boron-arsenic systems by Michael, with some assistance from Richard Jouet, (see publication 4 below) resulted in the isolation and structural characterization of $\text{I}_3\text{B}\bullet\text{As}(\text{SiMe}_3)_3$, as well as the isolation of X-ray quality crystals of the $\text{Cl}_3\text{B}\bullet\text{As}(\text{SiMe}_3)_3$ and $[\text{I}_2\text{BAs}(\text{SiMe}_3)_2]_2$ (preparations previously reported in the 1996 "Interim Technical Report"). Additional studies by Richard involving $(\text{Et}_2\text{O})_2\text{Li}(\mu\text{-P}(\text{SiMe}_3)_2)_2\text{GaH}_2$ showed that (1) in a 1:1 mole ratio reaction with BCl_3 , both known $[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_3$ and new $\text{Cl}_2\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{BH}_2$ were produced, (2) from a 1:1.25 mole ratio reaction with MeBCl_2 , both $(\text{Me}_3\text{Si})_2\text{P}(\text{H})\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{GaH}_2$ and $(\text{Me}_3\text{Si})_2\text{P}(\text{H})\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{B}(\text{H})\text{Me}$ could be isolated (cocrystallized in a 1:1 mole ratio), and (3) the 1:2.8 mole ratio reaction with MeBCl_2 afforded $(\text{Me}_3\text{Si})_2\text{P}(\text{Cl})\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{B}(\text{H})\text{Me}$ as the only species to crystallize from solution. In addition, he found that the known trimer $[\text{H}_2\text{GaP}(\text{SiMe}_3)_2]_3$ was the only compound that could be isolated and characterized from the 1:1 mole ratio reactions of $(\text{Et}_2\text{O})_2\text{Li}(\mu\text{-P}(\text{SiMe}_3)_2)_2\text{GaH}_2$ with (1) Me_2BBr and (2) $\text{H}_2\text{BCl}\bullet\text{SMe}_2$.

Publications Describing Results Obtained

1. M. S. Lube, R. L. Wells, and P. S. White, "Preparation and Characterization of Halogen-Boron-Phosphorus Compounds; X-ray Crystal Structures of $[\text{X}_3\text{B}\bullet\text{P}(\text{SiMe}_3)_3$ and $[\text{X}_2\text{BP}(\text{SiMe}_3)_2]_2$ ($\text{X} = \text{Cl}, \text{Br}$)", *Inorg. Chem.* **1996**, 35, 5007.
2. M. S. Lube, R. L. Wells, and P. S. White, "Reactions of Boron Trihalides with Tris(trimethylsilyl)arsine and Lithium Bis(trimethylsilyl)arsenide; X-ray Crystal Structures of $[\text{X}_2\text{BAs}(\text{SiMe}_3)_2]_2$ ($\text{X} = \text{Cl}, \text{Br}$)", *Main Group Metal Chemistry* **1996**, 19, 733. (invited manuscript)
3. M. S. Lube, R. L. Wells, and P. S. White, "Synthesis, Characterization, and X-ray Crystal Structures of the Boron-Antimony adducts $\text{X}_3\text{B}\bullet\text{Sb}(\text{SiMe}_3)_3$ ($\text{X} = \text{Cl}, \text{Br}, \text{I}$)", *J. Chem Soc., Dalton Transactions* **1997**, 285.
4. M. S. Lube, R. J. Jouet, R. L. Wells, P. S. White, and V.G. Young, Jr., "Further Investigations into the Synthesis and Characterization of Halo-Boron-Arsenic Compounds: X-ray Crystal Structures of $\text{X}_3\text{B}\bullet\text{As}(\text{SiMe}_3)_3$ ($\text{X} = \text{Cl}, \text{I}$) and $[\text{I}_2\text{BAs}(\text{SiMe}_3)_2]_2$ ", *Main Group Chemistry* **in press**.

Presentations at Meetings Describing Results Obtained

1. M. S. Lube, R. L. Wells, and P. S. White. "Syntheses, Characterization and Thermolyses of Potential Precursors to Boron Antimonide." 212th National Meeting of the American Chemical Society, August 25-29 1996, Orlando, FL.
2. R. J. Jouet, R. L. Wells, and A. L. Rheingold, "Investigations into the Reactivity of the Lithium Pnictidogallates: Reactions of $(\text{Et}_2\text{O})_2\text{LiE}(\text{SiMe}_3)_2\text{Ga}(\text{H})_2\text{E}(\text{SiMe}_3)_2$ ($\text{E} = \text{P}, \text{As}$) with BCl_3 and R_2BCl ($\text{R} = \text{H}, \text{Me}$)", 111th Sectional Conference of the North Carolina Section of the American Chemical Society, Durham, NC, April 5, 1997. (* should have read "..... with BCl_3 , $\text{H}_2\text{BCl}\bullet\text{SMe}_2$ and MeBCl_2 ")
3. R. J. Jouet, R. L. Wells, J. F. Janik, and P. S. White, "Lithium Pnictidogallate Reactivity: Reactions of $(\text{Et}_2\text{O})_2\text{Li}[\mu\text{-E}(\text{SiMe}_3)_2]_2\text{GaH}_2$ ($\text{E} = \text{P}, \text{As}$) with Group 13 Halides", Abstracts of Papers, 214th American Chemical Society National Meeting, September 7-11, 1997, INOR 351, Las Vegas, NV.